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(54) Name of the invention: Melt Blow Method and Melt Blow Equipment

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Description of the Invention

1. Name of the Invention

Melt Blow Method and Melt Blow Equipment

2. Scope of the Claims

1. Melt blow method characterized by the fact that it is a melt blow method where from a die nozzle, which has a cross section that is a triangular shape with two sides being equal and that has a large number of die orifices (1), which are provided practically on the tip part (A) of the bilateral triangular shape, molten polymer material is jetted out, and by jet propulsion of heated gas in the vicinity of the edges of these orifice openings, the above described sent out polymer is dragged and made to be fine; and it is a method where the above described polymer material is a condensation type polymer material, and also, where the above described die nozzle tip part (A) is a surface that has a width (W), which in the range of 0.10 ~ 0.20 mm.
2. Melt blow method according to the above described Claim 1 of the present invention characterized by the fact that the above described condensation type polymer material is polyester, polyamide.
3. Melt blow method according to the above described Claim 1 of the present invention characterized by the fact that the temperature of the above described heated gas is in the range if 320 ~ 370°C.
4. Melt blow method according to the above described Claim 1 of the present invention characterized by the fact that the pressure of the above described heated gas is in the range of 1.5 ~ 6.0 kg/cm².

5. Melt blow equipment characterized by the fact that it is a melt blow equipment that has a melt blow die, and the above described melt blow die has a cross section with a shape of triangle with two equal sides, and it has a die nozzle which has a large number of orifices that are placed practically on the tip part of the triangular shape with two equal sides, and it has a two fluid slits, which are formed by a lip plate that is bent to the side of the orifices, and it is an equipment where the above described die nozzle tip part is a surface that has a width which is in the range of 0.10 ~ 0.20 mm, and the die nozzle tip part is positioned on the outer side at a distance that is only 0.01 ~ 0.40 mm from the two lip plate tip part, and the diameter of the die orifice ϕ is in the range of 0.15 ~ 0.50 mm, and also, the width (X) of the fluid slits satisfies the relationship $r/4 \leq X \leq 5/4$.
6. Melt blow equipment according to the above described Claim 5 of the present invention, characterized by the fact that the tip angle of the above described triangular shape with two equal sides, that is formed as the cross sectional surface of the above described die nozzle, is in the range of 45° ~ 90°.
7. Melt blow equipment according to the above described Claim 5 of the present invention, characterized by the fact that the above described die nozzle tip part is a practically a flat surface, and also, the width of this flat tip surface is in the range of 0.12 ~ 0.16 mm.
8. Melt blow equipment according to the above described Claim 5 of the present invention, characterized by the fact that the diameter of the above described die orifice is in the range of 0.20 ~ 0.40 mm.

3. Detailed Explanation of the Invention

The present invention is an invention about a melt blow method for the manufacturing of good, extremely fine fiber web that has a high tensile strength and also where there is no generation of polymer gels, from condensation type polymer materials, where this manufacturing is stable over prolonged periods of time, and it is also an invention about its equipment.

The fundamental melt blow equipment and method have been disclosed and presented in the report in Industrial and Engineering Chemistry, volume 48, number 8 (page 1342 ~ 1346), 1956. In the description reported in the Japanese Patent Application Laid Open Number Showa 49-48921, equipment has been disclosed where the die nozzle tip part has been sharpened and not only that but also, where the die nozzle tip part (A) is protruding forward from the lip plate tip part (B) at a distance in the range of 0.13 ~ 0.25 mm.

However, in the case of the above described type of equipment according to the previous technology, the width (W) of the die nozzle tip part is no more than 0.07 mm or less, and it has an extremely sharp shape like a blade, and because of that during the handling or during the assembly of the spinning opening, it has been easy for debris to adhere on the tip part. For example, in order to manufacture good quality web with little generation of polymer gels (balls), it is necessary that the adjustment of the width (X) of the fluid slit is constantly adjusted in the width direction (the direction of the length of the die) at the order of 1/100 mm. However, if for example, this adjustment is conducted as a gap gauge is pushed in the space between the die nozzle (2) and the lip plate (3), in the cases when somehow through the gap gauge a debris is adhered on the die nozzle tip part (including the orifice openings), and it is bent, the adjustment is lost. Also, regarding the adjustment of the position of the die nozzle tip part (A) and the lip plate tip part (B) (the distance Y), the same way an extremely fine adjustment is required in the direction of the width, and at the time when this position is measured, for example, it is necessary that the tip of the dial gauge is facing against the tip part (A) of the die nozzle. And in the case when the width of the tip part is no more than 0.07 mm or less, this measurement is quite difficult, and not only that but also, there is a significant amount of adhered debris on the die nozzle tip part at the time of the measurement. If such debris is present even on one location, from this part a relatively large polymer gel (ball) is generated, and the value as a manufactured product is lost. In the case when a long die (multihole die nozzle) is used in order to manufacture a large width web, this problem becomes especially significant. Because of that, the manufacturing of a good quality web at a large width (especially, a width of at least 1000 mm and above) has been very difficult in practice. Also, in the case when during the time of the fiber spinning, polymer material etc., are adhered onto the surface of the spinning opening, it is difficult to wipe the tip part so that the debris is eliminated, and there has also been the problem that the continuous fiber spinning for prolonged periods of time, has not

been possible. Especially, in the case of the equipment according to the previous technology, specifically the manufacturing of good quality web with little generation of polymer gels, from polyester, polyamide etc., condensation type polymer materials, is relatively difficult, and also the problem has been observed that the tensile strength of the web has been low. If there are such problems the applications are very limited, and especially, it becomes unusable as a base fabric for raised nap synthetic leather material. Namely, the generation of polymer gels (balls) deteriorates the surface appearance and the touch feel, and also, the tensile strength of the fiber is low, and because of that the tensile strength of the manufactured product is low, and not only that, but also, the friction durability degree, and especially, the humidity friction durability degree becomes changed.

The authors of the present invention have conducted rigorous research in order to improve such problems, and previously, according to the description reported in the Japanese Patent Application Laid Open Number Showa 53-9355, they have invented a die where the tip part of the die nozzle is positioned at a distance within 0 ~ 0.4 mm from the two lip plate tip parts. Then, especially, research was accumulated and as a result of that it was observed that especially it is effective in obtaining from condensation type polymers like, polyester or polyamide, a good quality extremely fine fiber large width web material, where the tensile strength is large, and not only that but also, where there is no generation of polymer balls. And based on that the present invention was achieved.

And namely, the present invention is an invention about a melt blow method characterized by the fact that it is a melt blow method where from a die nozzle, which has a cross section that is a triangular shape with two sides being equal and that has a large number of die orifices (1), which are provided practically on the tip part (A) of the bilateral triangular shape, molten polymer material is jetted out, and by jet propulsion of heated gas in the vicinity of the edges of these orifice openings, the above described sent out polymer is dragged and made to be fine; and it is a method where the above described polymer material is a condensation type polymer material, and also, where the above described die nozzle tip part (A) is a surface that has a width (W), which in the range of 0.10 ~ 0.20 mm;

and it is also an invention about a melt blow equipment characterized by the fact that it is a melt blow equipment that has a melt blow die, and the above described melt blow die has a cross section with a shape of triangle with two

equal sides, and it has a die nozzle which has a large number of orifices that are placed practically on the tip part of the triangular shape with two equal sides, and it has two fluid slits, which are formed by a lip plate that is bent to the side of the orifices, and it is an equipment where the above described die nozzle tip part is a surface that has a width which is in the range of 0.10 ~ 0.20 mm, and the die nozzle tip part is positioned on the outer side at a distance that is only 0.01 ~ 0.40 mm from the two lip plate tip part, and the diameter of the die orifice ϕ is in the range of 0.15 ~ 0.50 mm, and also, the width (X) of the fluid slits satisfies the relationship $r/4 \leq X \leq 5/4$.

The present invention will be described in further details based on the diagrams presented.

Figure 1 is a diagram of the longitudinal direction sectional view of the die according to the present invention, at the maximum orifice diameter position. The die consists of the die nozzle (2), which has a sectional surface with a triangular shape where the two sides are equal, and the attached on its both sides two lip plates (3), that are used for the adjustment of the slit for the fluid. It is preferred that the tip angle of the sectional surface of the die nozzle, that has a triangular shape where the two sides are equal, is in the range of 45° ~ 90°, and especially, according to the present invention it is preferred that it be in the range of 55° ~ 75°. On this tip part (A), that has a sectional surface with a triangular shape where the two sides are equal, a surface with a width (W), which is in the range of 0.10 ~ 0.20 mm, is formed, and it is a good option if this edge surface is a surface that has a large number of protrusions and indentations and it is also a good option if it is a curved surface, however, it is preferred that it is practically a flat surface, and not only that, but also, it is preferred that the width (W) of this edge flat surface be in the range of 0.12 ~ 0.16 mm. In this die nozzle a large number of orifices are drilled. Regarding the placement of these orifices, it is best if the orifice center coincides with the crossing point (C) of the two sides of the sectional surface of the die nozzle that has a triangular shape where the two sides are equal, however, it is also a good option if the center is at a distance from this crossing point (C) to within the radius of the orifice. The orifice diameter is larger than the width (W) of the tip part.

On both sides of the die nozzle the lip plates (3) are positioned. In the case of the lip plates, their edge part (B) becomes a sharp angle knife edge. The width of the tip of this edge is no more than 0.20 mm, or less, and especially, it is preferred that it be no more than 0.10 mm or less. By the outer surface

of the sectional surface of the die nozzles (2), that has a triangular shape where the two sides are equal, and by the surface of the lip plates, slits are formed one on the right and the left side of the die nozzle correspondingly, that are bent along the orifice line, and that are used for long fluids.

Regarding the above described two fluid slits that are formed at the outer surface, it is preferred that they be parallel. Also, these lip plates are adjusted by bolts etc., and by that adjustment it is possible to change freely the gap between these fluid slits.

As the condensation type polymer material, there are the following materials: polyethylene terephthalate, polybutylene terephthalate, etc., polyesters, Nylon 6, Nylon 66, etc., polyamides. However the materials that can be used are not limited to these. Also, it is a good option if these are copolymer materials, and it is also a good option they are materials obtained as two types of polymers are blended. According to the present invention, polyethylene terephthalate is especially preferred.

However, in the case when the die according to the present invention is used for polypropylene, polystyrene, etc., adduct polymer materials, there is a generation of polymer gels (balls) and it is difficult to obtain a good web.

As the heated gas that is jet propelled from the fluid slits, it is possible to use steam, air etc. When the gas temperature is in the range of 300 ~ 390°C, and especially in the range of 320 ~ 370°C, it is preferred because of the fact that the decrease of the web viscosity is small. Also, regarding the gas pressure that is found at the fluid slit parts, it is at least 1.2 kg/cm² or higher, and especially, it is preferred that it is in the range of 1.5 ~ 6.0 kg/cm², because of the fact that there is little generation of polymer gels and it is possible to obtain very fine fibers where the average fiber diameter is in the range of 1.0 ~ 6.0 microns.

According to the present invention it is especially important that the tip part (A) of the die nozzle is a surface that has a width that is in the range of 0.10 ~ 0.20 mm, and preferably, in the range of 0.12 ~ 0.16 mm. In the case of such a die, the adhesion of debris onto the tip part during use, is small, and besides that, for example, even if some debris has adhered, it is difficult to generate a disturbance in the gas flow, and the fiber spinning properties are almost unchanged. Also, because the slit width adjustment and the determination of the position of the die nozzle tip part and the lip plate tip

part, are conducted easily and also precisely, it is possible to obtain homogeneous wide width web material. Also, the polymer adhesion in the area around the spinning opening and especially, on the die nozzle tip part, is small, and because of that under high gas pressure conditions, a fiber spinning process that is stable over prolonged periods of time, becomes possible, and for example, even if there is polymer adhesion, it is possible to be restored to the correct state by wiping. In the obtained web material there is extremely little generation of polymer gels, and not only that but also the web tensile strength is also high. However, in the case when the width (W) of the tip part has exceeded 0.20 mm, the generation of this polymer gel is significantly increased, and a good quality manufactured product is not obtained. The above described results are especially confirmed in the case of condensation type polymer materials like polyesters.

Regarding the term polymer gel (ball) used throughout the description of the present invention, it is the spherical shaped polymer that has a diameter that is 10 ~ 500 times the diameter of the fiber that forms the structure of the web, or it is the swollen shape polymer, that is generated at the edge part or the internal part of the fiber. There are many cases when these polymer gels are extremely small and they cannot be observed by the naked eye. It is possible to detect them by observation using a microscope, or it is possible for them to be detected as the web in the state as it is or, after it has been pressed, calendered, treated by squeezing, or by using other treatment measures, the fiber density is increased and after that it is colored. If a large number of these polymer gels are present, the application sphere is largely limited, and especially, the material becomes unusable as a base fabric for synthetic leather material.

In the case according to the present invention, the die nozzle tip part (A) is positioned on the outer side (the downward direction according to Figure 1) from the two lip plate tip parts (B) only at a distance Y, that is in the range of 0.01 ~ 0.40 mm. Also, relative to the orifice diameter ϕ being in the range of 0.15 ~ 0.50 mm, and preferably in the range of 0.20 ~ 0.40 mm, it is especially preferred that the width (X) of the fluid slit be within the range of $r/4 \leq X \leq 5/4 r$. If a melt blow process is conducted by using a die which satisfies simultaneously this relationship, even under a wide range of fluid pressure conditions, in the range of 1.5 ~ 6.0 kg/cm²G, it becomes possible to stably manufacture over prolonged period of time, a good quality web material that does not have polymer gels. And especially, in the case of polyesters like a polyethylene terephthalate material according to the

previous technology it has been difficult to manufacture a good quality web with no generation of polymer gels, since it has been necessary to have a melt viscosity of no more than 300 Poases or less, because of an extreme degree of thermal destruction. However, according to the present invention, the die temperature is decreased and even at a high melt viscosity state of up to 2,000 Poases, it is possible to obtain a good quality web material with no generation of polymer gels, and because of that it becomes possible to manufacture a web with a high tensile strength.

On the other hand, in the case when the distance (Y) has exceeded 0.40 mm, the polymer that exits from the orifice is a high rate high pressure fluid and it is not very efficiently melted, and the polymer is not transformed into extremely fine fibers and there is a generation of a large number of polymer gels inside the web material. On the contrary, if the tip part (A) of the die nozzle is made to be the same surface as that of the two tip parts (B) of the lip plates, or higher, or if somehow it is made to be on the inner side (according to the presented in Figure 1, the A surface is upward from the surface B), it becomes difficult to have a continuous conversion for a time period exceeding 500 hours, and not only that, but also, the strength of the obtained extremely fine fiber web becomes somewhat low. Also, in the case when the width (X) of the fluid slit is smaller than $r/4$, on the lip front surface and on the die tip part polymer adhesion is observed, and on the contrary, in the case when X is larger than $5/4 r$, it is necessary to use extremely large gas flow rates and not only that, but also, the fluid energy that is imparted to the molten polymer material is excessive, and the viscosity of the produced web is decreased and breaks of the fibers are generated, and as a result from that the web strength is decreased and there is a generation of polymer gels.

In the case of the present invention, the tip part of the die nozzle becomes a surface in the range of 0.10 ~ 0.20 mm, and because of that, the fluid slit width, and the determination of the positions of the die nozzle tip part and the lip plate tip parts, can be conducted with a good accuracy in the width direction (the direction of the length of the die), and as a result from that it is possible to obtain a wide width manufactured product which is homogeneous even in the width direction (for example, there is a small distribution of the partial polymer gel generation or the weight per surface area, that is found in the direction of the width). Also, it is possible to conduct wiping of the surface of the spinning openings at the time of the fiber spinning, and a continuous stable fiber spinning over prolonged period

of time, becomes possible. Not only that, but also, because of the fact that the debris adhesion on the die nozzle tip part, during the exploitation, is small, it is also effective from a protection point of view and it is also economical.

The finer fiber web made of condensation type polymer material, that is obtained according to the present invention, can be used in different applications areas such as filters, separators, etc., however, especially, it can be used preferably as a foundation fabric material used for synthetic leather because it is a high quality product that has a high tensile strength. And namely, by mutually crossing the extremely fine fibers and after that making a rubber properties possessing elastic type polymer material like polyurethane present in the gaps between the fibers, and raising the nap on the surface, and coloring, it is possible to obtain a high grade suede type or nupak type synthetic leather material, that has a smooth fine fiber feel to it. The fact that it can be stated that the color durability degree, and especially the humid friction color durability degree is extremely good, is also an important characteristic of the present invention.

Here below the present invention will be especially explained in further details by presenting practical implementation examples. Moreover, the web tensile strength that is described according to these practical implementation examples, is the value (g/cm) that was measured as a web with 100 g/m² weight per surface area, and a web width of 1.0 cm, and by using a Tensiron with a clamp distance of 1.0 cm, Also, the web viscosity is a value that has been measured at a temperature of 35°C in ortho-chlorophenol.

Practical Example 1

According to the presented in Figure 1, relative to a die nozzle (2), that has a tip angle of 60°, and where the tip part surface is a flat surface and the width (W) of that surface is 0.12 mm, and where on this tip part 1,000 orifices with a diameter ϕ of 0.30 mm ϕ , have been pierced separated at a pitch of 1 mm, the two lip plates (3) that have a tip part angle of 60°, are set so that the width (X) of the fluid slit is 0.25 mm and so that the die nozzle tip part (A) is positioned on the outside part (the distance (Y), according to the diagram presented in Figure 1) at 0.10 mm from the two lip plate tip parts (B). The maximum adjustment variation that occurs in the width direction (the length direction of the die) of the above described X and Y, was no more than 0.05 mm, or less.

Polyethylene terephthalate ($\eta_{sp}/c = 0.73$) was melted at a temperature of 295°C, and it was jet propelled out through the above described orifices at a jet propelled amount of 0.15 g/minute/orifice, and steam was propelled through the slits (4) at 2.7 kg/cm²G, and a temperature of 340°C. By the positioned downstream collection device (not shown in the figure) a web with a width of 1,000 mm was made, and collected and it was continuously wound. A continuous transformation was conducted for a period of 750 hours, and despite that during that period of time there was almost no staining of the surface of the spinning opening. The obtained web had extremely fine fibers with an average fiber diameter of 2.5 microns, and despite that there were no polymer gels, and the unevenness in the weight per surface area in the direction of the width of the web material, was 7 %, and it was an extremely good web material. Also, the viscosity η_{sp}/c of the web was 0.62, and the degree of destruction was also small and the strength of the web material was 820 g/cm. When this web was subjected to a crossing treatment and then according to the usually used methods polyurethane material was filled in the interstices, it was possible to obtain an extremely good synthetic leather material. The humid friction coloring durability degree of this synthetic leather material was 2 ~ 3 class.

Practical Example 2

According to the presented in Figure 1, relative to a die nozzle (2), that has a tip angle of 80°, and where the tip part surface is a flat surface and the width (W) of that surface is 0.20 mm, and where on this tip part 1,000 orifices with a diameter ϕ of 0.40 mm ϕ , have been pierced separated at a pitch of 1 mm, the two lip plates (3) that have a tip part angle of 60°, are set so that the width (X) of the fluid slit is 0.40 mm and so that the die nozzle tip part (A) is positioned on the outside part (the distance (Y), according to the diagram presented in Figure 1) at 0.10 mm from the two lip plate tip parts (B). The maximum adjustment variation that occurs in the width direction (the length direction of the die) of the above described X and Y, was no more than 0.05 mm, or less.

Polyethylene terephthalate ($\eta_{sp}/c = 0.73$) was melted at a temperature of 320°C, and it was jet propelled out through the above described orifices at a jet propelled amount of 0.20 g/minute/orifice, and steam was propelled through the slits (4) at 2.0 kg/cm²G, and a temperature of 370°C. By the positioned downstream collection device (not shown in the figure) a web

with a width of 1,000 mm was made, and collected and it was continuously wound. A continuous transformation was conducted for a prolonged period of time, and despite that during that period of time there was almost no staining of the surface of the spinning opening. Also, the viscosity η_{sp}/c of the web was 0.60, and the strength of the web material was sufficiently high.

Practical Example 3

According to the presented in Figure 1, relative to a die nozzle (2), that has a tip angle of 60° , and where the tip part surface is a flat surface and the width (W) of that surface is 0.15 mm, and where on this tip part 1,000 orifices with a diameter ϕ of 0.30 mm ϕ , have been pierced separated at a pitch of 1 mm. And also, regarding the lip plates, they set so that the die nozzle tip part (A) is positioned on the outside part (the distance (Y), according to the diagram presented in Figure 1) at a distance of 0.25 mm from the two lip plate tip parts (B), and the width (X) of the fluid slit was set according to the presented in Table 1. Polyethylene terephthalate ($\eta_{sp}/c = 0.73$) was jet propelled under the same conditions as those described according to the reported above Practical Example 1, and it was melt blown and made into a web material and wound.

As it is clear from the presented in table 1, if the slit width (X) is within the range of 0.08 ~ 0.37 mm, namely, if it is within the range of $r/4 \sim 5/4 r$, it is understood that it is possible to manufacture over prolonged period of time a high quality extremely fine fiber web material, where there is no generation of polymer gels, where the decrease of the viscosity is small and the strength is high.

Table 1

Slit Width (X) [mm]	Staining of the surface of the spinning opening occurring during a continuous conversion (750 hour period)	Generation of Polymer gels in the manufactured product web	Average fiber diameter (microns)	Web strength (g/cm)	Web viscosity (η_{sp}/c)	Overall Evaluation
0.05	Not good	Many	4.0	860	0.66	X
0.08	Good	Little	3.5	855	0.63	O
0.20	Very good	Not present	2.5	820	0.60	O
0.37	Very good	Extremely little	2.5	810	0.58	O
0.40	Good	Many	3.5	680	0.48	X

Practical Example 4

According to the presented in Figure 1, the tip angle was 50°, and the tip part surface is a surface where the width (W) of that surface is 0.10 mm, and where on this tip part 1,000 orifices with a diameter ϕ of 0.20 mm ϕ , have been pierced. And also, regarding the lip plates, the fluid slit width (X) was made to be 0.28 mm, and especially, they were set so that the die nozzle tip part (A) is positioned on the outside part (the distance (Y), according to the diagram presented in Figure 1) at a distance of 0.35 mm from the two lip plate tip parts (B).

Nylon 6 was melted at a temperature of 290°C, and it was jet propelled out through the above described orifices at a jet propelled amount of 0.18 g/minute/orifice, and steam was propelled through the slits (4) at 3.5 kg/cm²G, and a temperature of 330°C. A continuous transformation was conducted for a prolonged period of time, and despite that during that period of time there was no staining of the surface of the spinning opening, and a

very good, extremely fine fiber web material (average fiber diameter 1.5 microns) was obtained. Also, the web viscosity was sufficiently high, and it was a web that had a high strength.

Reference Example 1

According to the presented in Figure 1, the tip angle was 60°, and the tip part surface is a surface where the width (W) of that surface is 0.05 mm, and where on this tip part 1,000 orifices with a diameter ϕ of 0.30 mm ϕ , have been pierced separated at a pitch of 1 mm. And also, regarding the lip plates, the fluid slit width (X) was made to be 0.30 mm, and especially, they were set so that the die nozzle tip part (A) is positioned on the outside part (the distance (Y), according to the diagram presented in Figure 1) at a distance of 0.25 mm from the two lip plate tip parts (B). However, the adjustment was somewhat difficult, and the maximum adjustment deviation in the width direction (the direction of the length of the die) was 0.10 mm.

Polyethylene terephthalate ($\eta_{sp}/c = 0.73$) was melt blown under the same conditions as those described according to the reported above Practical Example 1, however, from 300 hours after the beginning of the continuous conversion process, the polymer staining on the surface of the spinning opening and on the lip edges, was significant, and it was not possible to continue the fiber spinning process. In the web material that was obtained in the latter half of the fiber spinning process, the generation of polymer gels was especially high, and it was also a material where the web strength was very low at 650 g/cm. By using this web material and following the same procedures as those described in the Practical Example 1, a raised nap synthetic leather material was obtained, however, it was a material where the feel of the front surface was rough.

4. Simple Explanation of the Figures

Figure 1 is a sectional view diagram of the melt blow die in the die orifice maximum diameter position, according to the equipment of the present invention.

- 1.....die orifice
- 2.....die nozzle
- 3.....lip plate
- 4.....fluid slit

A.....die nozzle tip part
B.....lip plate tip part
C.....die nozzle extrapolation lines crossing
point
X, Y.....distance
r.....diameter
W.....width

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⑭ メルトブロー法およびメルトブロー装置

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明 細 書

1. 発明の名称

メルトブロー法およびメルトブロー装置

2. 特許請求の範囲

1. 断面が二等辺三角形であり、二等辺三角形の實質上の尖端部(A)に配設された多数のダイオリフィス(1)を有するダイノーズから熔融ポリマーを吐出し、このオリフィスの開口端近傍に加熱ガスを噴射することにより前記吐出ポリマーを牽引融化するメルトブロー法において、前記ポリマーが縮合系ポリマーであり、かつ前記ダイノーズ尖端部(A)が0.10～0.20 mmの幅(W)を有する面であることを特徴とするメルトブロー法。

2. 前記縮合系ポリマーがポリエステル、ポリアミドであることを特徴とする特許請求の範囲第1項記載のメルトブロー法。

3. 前記加熱ガスの温度が320～370℃であることを特徴とする特許請求の範囲第1項記載のメルトブロー法。

4. 前記ガス圧力が1.5～6.0 kg/cm²であるこ

とを特徴とする特許請求の範囲第1項記載のメルトブロー法。

5. メルトブローダイを有するメルトブロー装置において、前記メルトブローダイが断面が二等辺三角形であり、二等辺三角形の實質上の尖端部に配設された多数のダイオリフィスを有するダイノーズと、オリフィスの列にやつてリップ板によって形成された2つの流体スリットとを有し、前記ダイノーズ尖端部が0.10～0.20 mmの幅を有する面であり、ダイノーズ尖端部は2つのリップ板尖端部から0.01～0.40 mmの距離だけ外側に位置しており、ダイオリフィスの直径(r)が0.15～0.50 mmであり、かつ、流体スリットの幅(X)が $r/4 \leq X \leq 5/4 r$ の関係を満足することを特徴とするメルトブロー装置。

6. 前記ダイノーズの断面における前記二等辺三角形の尖端角度が45°～90°であることを特徴とする特許請求の範囲第5項記載のメルトブロー装置。

7. 前記ダイノーズ尖端部が實質的な平面であり、かつ、この尖端平面の幅が0.12～0.16 mm

であることを特徴とする特許請求の範囲第5項記載のメルトブロー装置。

8. 前記ダイオリフィスの直径が0.20~0.40mmであることを特徴とする特許請求の範囲第5項記載のメルトブロー装置。

3. 発明の詳細な説明

本発明は、縮合系ポリマーから、強力が強くしかもポリマー玉を発生させずに良質な極細繊維ウェブを長時間安定に製造するメルトブロー法およびその装置に関する。

メルトブローについては、インダストリアル・アンド・エンジニアリング・ケミストリー (Industrial and Engineering Chemistry 48巻、第8号(p 1342~1346)、1956年に基本的な装置及び方法が開示されている。特開昭49-48921号公報には、ダイノーズ先端部をとがらせ、しかもダイノーズ先端部(A)がリップ板先端部(B)より0.13~0.25mm前に出した装置が示されている。

しかしながら、前記した様な従来の装置は、ダ

(3)

比較的大きなポリマー玉が発生し、製品としての価値を失ってしまう。この問題は、広巾ウェブを得るための長いダイ(多ボールダイノーズ)を用いた場合に特に大きな問題となる。このため、広巾(特に1000mm巾以上)の良質なウェブを得ることには実質上著しく困難であった。また、紡糸時に紡口面にポリマー等が付着した場合、先端部をキズ付けずにワイピングすることが困難であり、長時間の連続紡糸が出来ないという問題もあった。更に、従来の装置は、特にポリエステル、ポリアミドの様な縮合系ポリマーからポリマー玉の発生が少ない良質なウェブを得ることが比較的困難であり、ウェブ強力が低いという問題も見い出された。この様な問題があると用途が著しく制限されてしまい、特に起毛させた人工皮革用織布として用いられなくなる。すなわち、ポリマー玉の発生は、表面の見栄え、タッチを損ねるし、また繊維強力が低いことは、製品の強力を低くするのみならず表面の摩擦堅牢度、特に湿摩擦堅牢度を悪化させることになる。

(5)

イノーズ先端部巾(W)が0.07mm以下と極めて尖ったナイフの刃の如きの形状をしているため、取扱い時や紡口組立時に重要な先端部をキズ付け易いものであった。たとえば、ポリマー玉の発生が少ない良質なウェブを製造するためには、流体スリット巾(X)の調整を1/100mmのオーダーで巾方向(ダイの長さ方向)で一定に調整する必要があるが、この調整を例えばすきまゲージをダイノーズ(2)とリップ板(3)との間に挿入して行くと、すきまゲージによりどうしてもダイノーズ先端部(オリフィス孔を含む)をキズ付けたり、曲げたり、場合によっては欠けたりしてしまう。また、ダイノーズ先端部(A)とリップ板先端部(B)との位置(距離Y)の調整も同様に巾方向で精密に調整する必要があるが、この位置の測定を行う際、例えばダイヤルゲージの先端をダイノーズ先端部(A)に当てる必要があり、先端部巾が0.07mm以下の場合はこの測定が著しく困難であるし、しかも測定時にダイ先端部をキズ付けることが多い。この様なキズが1ヶ所でも存在すると、その部分から

(4)

本発明者らは、この様な問題を改善すべく鋭意研究し、先に特開昭53-93555号にダイノーズ先端部幅が0.1~0.2mmであり、ダイノーズ先端部が2つのリップ板先端部から0~0.4mmだけ内側に位置せしめるダイを提案した。ここに、更に研究を重ねた結果、特にポリエステルやポリアミドの如き縮合系ポリマーからより強力が大きく、しかもポリマー玉の発生のない良質な極細繊維の広巾ウェブがより有利に得られることを見出し、本発明を完成した。

すなわち、本発明は、断面が二等辺三角形であり、二等辺三角形の頂上の先端部(A)に配設された多数のダイオリフィス(1)を有するダイノーズから熔融ポリマーを吐出し、このオリフィスの開口端近傍に加熱ガスを噴射することにより前記吐出ポリマーを牽引細化するメルトブロー法において、前記ポリマーが縮合系ポリマーであり、かつ前記ダイノーズ先端部(A)が0.10~0.20mmの幅(W)を有する面であることを特徴とするものであり、また、断面が二等辺三角形であり、二等辺

(6)

三角形の突質上の先端部(A)に配設された多数のダイオリフィス(1)を有するダイノーズ(2)と、オリフィスの列にそってリップ板(3)によって形成された2つの流体スリット(4)を有するメルトブローダイにおいて、ダイノーズ先端部(A)が0.10～0.20mmの幅(W)を有する面であり、ダイノーズ先端部(A)は2つのリップ板先端部(B)から0.01～0.40mmの距離(Y)だけ外側に位置しており、ダイオリフィスの直径(r)が0.15～0.50mmであり、かつ、流体スリットの幅(X)が $r/4 \leq X \leq 5/4r$ の関係であることを特徴とするものである。

本発明を図面によって詳細に説明する。

第1図は、オリフィスの最大直径位置における本発明のダイの縦方向断面図である。ダイは、二等辺三角形断面のダイノーズ(2)と、その両側に設けられた2つの流体スリット調節用リップ板(3)とからなる。ダイノーズの断面の二等辺三角形の尖端角は45°～90°が好ましく、特に55°～75°が本発明において好適である。この二等辺三角形の

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ト等によって調節することにより、この流体スリットはその間隙を自由に変えることができる。

融合系ポリマーとしては、ポリエチレンテレフタレート、ポリブチレンテレフタレートなどのポリエステル、ナイロン6、ナイロン6.6などのポリアミドがあげられるが、これらに限定されるものではない。また、共重合ポリマーであってもよく、2種のポリマーをブレンドしたものであってもよい。本発明では特にポリエチレンテレフタレートが好適である。

しかしながら、ポリプロピレン、ポリステレンなどの付加重合体では、本発明のダイを用いた場合、ポリマー玉が発生し、良好なウェブが得にくい。

流体スリットから噴射される加熱ガスとしては、スチーム、空気などが用いられる。ガス温度は300～390℃特に320～370℃がウェブの粘脱低下が少なく好ましい。また、流体スリット部におけるガス圧力は1.2 kg/cm²以上、特に1.5～6.0 kg/cm²がポリマー玉の発生が少ない平均値

(9)

先端部(A)には幅(W)0.10～0.20mmの面が形成されており、この先端面は多少の凸凹があっても、曲面であってもよいが、実質的な平面でありしかもこの先端平面の幅(W)が0.12～0.16mmであることが好ましい。このダイノーズには多数のオリフィスが穿孔されている。オリフィスの位置に関しては、オリフィスの中心がダイノーズの断面の二等辺三角形の2つの辺の交点上(C)に一致させるのが最もよいが、この交点(C)からオリフィスの半径以内の距離に中心があればよい。オリフィスの直径は先端部の幅(W)より大きい。

ダイノーズの両側にはリップ板(3)が位置している。リップ板はその先端部(B)が鋭角のナイフエッジになっている。このエッジの先端の巾は0.20mm以下、特に0.10mm以下が好ましい。ダイノーズ(2)の二等辺三角形の外周と、リップ板との面とでオリフィス列にそった長い流体用のスリットが、ダイノーズの左右にそれぞれ1つずつ形成される。前記2つの外面で形成される流体スリットは平行である方が好ましい。また、このリップ板をボル

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直径1.0～6.0mmの極細繊維が得られるので好ましい。

本発明において、ダイノーズ先端部(A)は0.10～0.20mm、好ましくは0.12～0.16mmの幅を有する面であることが特に重要である。この様なダイは取扱時に先端部をキズ付けることが少ない上に、たとえ多少キズ付けてもガス流の乱れが生じにくく紡糸性を悪化させることがほとんどない。また、スリット巾の調整やダイノーズ先端部とリップ板先端部との位置決めが容易に且つ精度良く行えるため、均一な広巾ウェブが得られる。また、紡口近傍、特にダイノーズ先端部のポリマー付着が少なく、高ガス圧力条件下では長時間安定に紡糸することが可能となり、例えばポリマー付着があってもワイピングにより正常な状態に復帰できる。得られたウェブはポリマー玉の発生が極めて少なく、しかもウェブ強度も高い。しかしながら、先端部巾(W)が0.20mmを超えた場合は、このポリマー玉の発生が著しく増し、良質な製品にならない。前述した効果は特にポリエステルの

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様な組合系ポリマーにおいて顕著に認められる。

本明細書でいうポリマー玉とは、ウェブ形成繊維の直径の10～500倍程度の直径を有する玉状ポリマーまたは繊維の端部や中間部に生成したコブ状ポリマーのことである。このポリマー玉は極めて小さく肉眼で見出すことができないものが多い。顕微鏡を用いて観察するか、または、ウェブをそのまま、もしくはウェブをプレス、カレンダー、交絡処理その他の手段によって繊維密度を高めたうえで染色することによって検知できる。このポリマー玉が多く存在すると、用途が大きく制限され、特に人工皮革用基布としては用いられなくなる。

本発明は、ダイノーズ尖端部(A)が2つのリップ板尖端部(B)から0.01～0.40mmの距離(Y)だけ外側(第1図において下方)に位置している。また、オリフィスの直径(r)が0.15～0.50mm、好ましくは0.20～0.40mmにおいて、流体スリットの巾(X)が $r/4 \leq X \leq 5/4 r$ の範囲にあることが特に好ましい。この関係が同時に満足されたダイ

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なるし、しかも得られた極細繊維ウェブの強度が幾分低くなる。また、加体スリット巾(X)が、 $r/4$ より小さい場合は、リップ表面やダイ尖端部にポリマーの付着が見られ、逆に、Xが $5/4 r$ より大きい場合には極めて大きなガス流量を用いる必要があるばかりでなく、熔融ポリマーに与える流体のエネルギーが多すぎ、生成したウェブの粘度の低下や繊維の切断が頻発し、その結果ウェブ強度の低下やポリマー玉の発生がある。

本発明は、ダイノーズ尖端部が0.10～0.20mmの面となっているため、流体スリット巾やダイノーズ尖端部とリップ板尖端部との位置決めが巾方向(ダイの長さ方向)で精度良く行うことができ、その結果、巾方向でも均一なウェブ巾方向における部分的なポリマー玉の発生や目付量の分布差が少ない)広巾製品が得られる。また、紡糸時に紡口面のワイピングを行うことができ、長時間の連続安定紡糸が可能となる。しかも、取扱時にダイ尖端部をキズ付けたりすることが少ないので保守の面でも有利であり経済的であ

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を用いてメルトブローすると、1.5～6.0 μm^2 と広範囲な流体圧力条件下においてもポリマー玉のない良質なウェブが長時間安定に製造することが可能となる。特にポリエチレンテレフタレート(PET)の如きポリエステルは、従来は極度の熱分解をさせることにより熔融粘度を300ポイズ以下にしなければポリマー玉の発生のない良質ウェブを製造することは困難であったが、本発明によれば、驚くべきことにダイ温度を低下させて500～2000ポイズまでの高熔融粘度の状態にしてもポリマー玉のない良質なウェブが得られるため、強力の高いウェブが製造可能となる。

一方、距離(Y)が0.40mmを超えた場合は、オリフィスから出たポリマーは高速高圧流体とあまり有効には接触しないで、ポリマーは極細繊維化されないばかりかウェブ中にポリマー玉が多く発生するようになる。逆に、ダイノーズ尖端部(A)が2つのリップ板尖端部(B)と同一面上にするか、又は幾分でも内側(第1図でA面がB面より上方)にすると、500時間を超えた連続運転が困難と

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る。

本発明により得られる組合系ポリマーの極細繊維ウェブはフィルター、セパレーター等の種々の用途に用いることができるが、特に強力が大きく高品質であるため人工皮革用基布として好適に用いることができる。すなわち、極細繊維相互を交絡させた後繊維間隙にポリウレタンのようなゴム状弾性重合体を介在させ、表面を起毛し、染色することにより、極細繊維の滑らかな風合をもつスウェードタイプやヌバックタイプの高級感のある人工皮革を得ることができる。この様にして得られた人工皮革の染色堅牢度、特に摩擦擦染色堅牢度が極めて良好であるということも本発明の大きな特長である。

以下に実施例を示して本発明を更に詳細に説明する。尚、実施例中ウェブの引張り強度は、ウェブ目付100 g/m^2 、ウェブ幅1.0cm、把持長1.0cmでテンシロンを用いて測定した値(g/cm)である。また、ウェブ粘度はオルトクロロフェノール中35℃における測定値である。

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実施例 1

第1図において、先端角度が 60° 、先端部面が幅(W) 0.12 mmの平面であり、この先端部に直径(r) 0.30 mmのオリフィスが1 mmピッチで一列に1000個並んで穿孔されているダイノーズ(2)に対して、先端角度が 60° の2枚のリップ板(3)を、流体スリットの幅(X)が0.25 mm及びダイノーズ先端部(A)が2つのリップ板先端部(B)から0.10 mm外側(第1図において距離(Y))に位置するようにしてセットした。前記XとYの巾方向(ダイの受さ方向)における調整バラツキは最大0.05 mm以下であった。

ポリエチレンテレフタレート($\eta_{sp}/c=0.73$)を295℃で溶融して前記オリフィスから吐出量0.15 g/分/オリフィスで吐出し、スリット(4)から吐力2.7 kg/cm²G、温度340℃でスチームを噴射した。ダイの下方に設置した集積装置(図がされていない)で巾1.000 mmのウェブとして集積して連続的に巻取った。750時間の連続運転を行なったが、その間紡口面の汚れはほとんどなかった。

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ら圧力2.0 kg/cm²G、温度370℃のスチームを噴射させた。長時間連続運転を行なったが紡口面の汚れはなく、しかもポリマー玉の発生のない良好な極細繊維ウェブが得られた。また、ウェブ粘度 η_{sp}/c は0.60でありウェブ強度も充分強いものであった。

実施例 3

第1図のダイにおいて、先端角度が 60° 、先端部面(A)が幅(W) 0.15 mmの平面であり、この先端部に直径(r) 0.30 mmのオリフィスを1 mmピッチで1列に1000個並べて穿孔した。またリップ板は、ダイノーズ先端部(A)が2つのリップ板先端部(B)から0.25 mm外側(距離Y)に位置するようにし、スリット幅(X)を表1に示す様にセットした。ポリエチレンテレフタレート($\eta_{sp}/c=0.73$)を実施例1と同様な条件下で吐出してメルトブローしてウェブとして巻取った。

第1表から明らかな様にスリット幅(X)が0.08~0.37 mmの範囲、即ち $r/4 \sim 5/4r$ の範囲内にあれば、ポリマー玉の発生がなく、粘度低

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た。得られたウェブは平均繊維径2.5 μ mの極細繊維であり、しかもポリマー玉がなく巾方向での目付斑も7%と極めて良好なウェブであった。また、ウェブの粘度 η_{sp}/c は0.62であり分解程度も少なくウェブ強度は、820 g/cmであった。このウェブを交絡処理後常法により繊維間隙にポリウレタンを充填させると極めて良質な人工皮革を得ることができた。この人工皮革の湿摩擦染色堅牢度は2~3級であった。

実施例 2

第1図において、先端角度が 80° 、先端部面が幅(W) 0.20 mmの平面であり、この先端部に直径(r) 0.40 mmのオリフィスが1000個並んで穿孔されているダイノーズ(2)に対して、流体スリット巾(X)が0.40 mm及びダイノーズ先端部(A)が2つのリップ板先端部(B)から0.05 mm外側(距離Y)にしてセットした。

ポリエチレンテレフタレート($\eta_{sp}/c=0.73$)を320℃で溶融して前記オリフィスから吐出量0.20 g/分/オリフィスで吐出し、スリット(4)か

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下が少く強度の高い良質な極細繊維ウェブが長時間製造可能なことがわかる。

第 1 表

スリット巾(X) (mm)	連続運転(750時間)における紡口面汚れ	製品ウェブ中のポリマー玉の発生	平均繊維径(μ m)	ウェブ強度(g/cm)	ウェブ粘度(η_{sp}/c)	総合評価
0.05	不良	多い	4.0	860	0.66	×
0.08	良	少ない	3.5	855	0.63	(○)
0.20	良好	なし	2.5	820	0.60	(○)
0.37	良好	極めて少ない	2.5	810	0.58	(○)
0.40	良	多い	2.5	680	0.48	×

実施例 4

第1図のダイにおいて、先端角度が 50° 、先端部面(A)が幅0.10 mmの面であり、この先端部に直径0.20 mmのオリフィスを1000個穿孔した。またリップ板は、流体スリット幅(X)を0.28 mmにし、更にダイノーズ先端部(A)が2つのリップ

08

板尖端部(B)から0.35mm外側(距離Y)にしてセットした。

ナイロン6を290°で溶融して前記オリフィスから吐出量0.18g/分/オリフィスで吐出し、スリット4から圧力3.5kg/cm²G、温度330℃のステームを噴射させた。長時間連続運転を行ったが紡口面の汚れはなく、しかもポリマー玉の発生のない良好な極細繊維ウェブ(平均繊維径1.5μm)が得られた。また、ウェブ粘度は充分高く、強力な大きいウェブであった。

比較例1

第1図において、尖端角度が60°、尖端部面(A)が幅0.05mmの面であり、この尖端部に直径(r)0.30mmのオリフィスを1mmピッチで1,000個穿孔した。またリップ板3、流体スリット幅(X)を0.30mmとし、更にダイノーズ尖端部(A)が2つのリップ板尖端部(B)より0.25mm外側(第1図において下方)にセットした。しかし、調整がかなり難しく、巾方向(ダイの長さ方向)での調整バラツキが最大0.10mmあった。

を実施例1と同様にしてメルトブローしたが、運転開始後300時間頃から紡口面及びリップ先端部面のポリマー汚れが著しく起り紡糸を続行することが出来なくなった。紡糸後半に付られたウェブはポリマー玉の発生が特に多く、ウェブ強度も0.50g/cmと低いものであった。このウェブを用いて実施例1と同様にして起毛人工皮革を作ったが、表面感触がザラザラしたものであった。

4. 図面の簡単な説明

第1図は本発明装置においてダイオリフィスの最大直径位置におけるメルトブローダ1の断面図である。

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|---------------|---------|
| 1-ダイオリフィス, | X, Y-距離 |
| 2-ダイノーズ, | r-直径 |
| 3-リップ板, | W-巾 |
| 4-流体スリット, | |
| A-ダイノーズ尖端部, | |
| B-リップ板尖端部, | |
| C-ダイノーズ外縁の交点, | |

第1図

